

EFFECTS OF CHEMICAL ACTIVATION PROCESS ON THE SURFACE MORPHOLOGY OF BROOM STICK -BASED ACTIVATED CARBON

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Abstract

The surface morphology of anode materials plays a crucial role in determining the electrochemical performance of supercapacitors. The porous structure and pore size of anode in supercapacitor facilitates the efficient ion transport and electrolyte diffusion. In this study, the surface morphology of broom stick based materials as an anode material of supercapacitor is investigated. Activated carbon (AC) from the broom stick was prepared by two different chemical activation processes. The structure of the broom stick AC was observed by scanning electron microscope (SEM). The pore structure and size depending on the activation temperatures in each method were investigated. It was found out that the structure and pore size of AC were different depending on the activation process and activated temperature.

Keywords: activated carbon, chemical activation, porous structure, pore size and scanning electron microscope (SEM)

Introduction

Surface morphology plays a crucial role in determining the electrochemical performance of supercapacitors. A larger surface area allows for more active sites for charge storage, and thus it can enhance the capacitance. Due to their nanometer size, nanostructured materials such as nanoparticles, nanowires, and nanosheets provide a high surface area. Hierarchical pore structures can improve both energy and power density (Libber *et al.*, 2024) in supercapacitor. The presence of different sizes of pores including micropore (<2 nm), mesopore (2–50 nm), and macropore (>50 nm) supports the efficient ion transportation and electrolyte diffusion in supercapacitor. Surface morphology can also influence the electrical conductivity of the electrode material in energy storage devices. Morphologies that provide structural integrity can withstand the stress of repeated charge-discharge cycles, enhancing the durability and lifespan of the supercapacitor⁴. The interaction between the electrode surface and the electrolyte is critical. A morphology that promotes better wettability and contact with the electrolyte can improve the overall electrochemical performance (Ling Miao *et al.*, 2010).

The pore size in supercapacitor electrodes significantly influences their electrochemical performance. Micropores provide a large surface area for charge storage, which is beneficial for increasing capacitance. However, very small micropores may restrict the movement of larger electrolyte ions, potentially limiting the rate of charge and discharge¹. For instance, well-aligned nanostructures can provide better pathways for electron transport, reducing resistance (Kian Keat Lee *et al.*, 2014). Morphologies that provide structural integrity can withstand the stress of repeated charge-discharge cycles, enhancing the durability and lifespan of the supercapacitor <https://pubs.rsc.org/en/content/articlehtml/2020/ma/d0ma00384k> (Akhil Pradiprao Khedulkar *et al.*, 2024). The interaction between the electrode surface and the electrolyte is

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critical. A morphology that promotes better wettability and contact with the electrolyte can improve the overall electrochemical performance (Kumar *et al.*, 2021).

In this study, activated carbon is synthesized from broom stick biomass for the purpose of using as electrode materials in supercapacitor. The surface morphology of activated carbon depending on different processes of chemical activation is presented.

Experimental Details

Synthesis of Activated Biochar Powder by One-Step Chemical Activation Method

Activated carbon (AC) is prepared from the raw materials of broom stick. Firstly, the raw broom sticks were washed with fresh water in order to remove any impurities and then they were cut into small pieces. After that, they were dried in the muffle furnace at 105 °C for 1 h to remove the moisture content in the raw of broom stick. For the activation process, 10 g of dry broom stick pieces were treated with 50 g of 6 M KOH solution in Muffle furnace at 600 °C and 700 °C for 1 h. After finishing the reaction, grayish pink colour of activated carbon was obtained. Then, as-prepared AC was washed with 0.2 M of HCL solution and distilled water (DW) by centrifugation until to neutralize the base residue. Afterwards, the pure AC was dried at 80 °C for a few hours until the moisture was completely removed. Finally, the grayish pink colour of AC powder was successfully obtained. The synthesis procedure of activated AC is shown in Figure 1.



Figure 1 Synthesis process of activated biochar powders by one-step chemical activation process

Synthesis of Activated Carbon Fiber by Two-Step Chemical Activation Method

In two-step method, the ash of broom stick was firstly prepared and then it was followed by activation process. 15 g of dry broom stick pieces were burned at 600 °C and 700 °C for 1 h inside the Muffle furnace and the ash was obtained. Subsequently, activation process was followed by adding 3.11 g of the ash into 50 g KOH (6 M) solution. The mixed solution was stirred for 3 h in order to increase porosity and surface area of ash. After that, the product was centrifuged with 0.4 M of HCL solution and DW for several times until to neutralize the KOH base residue. Then, the product was heated at 80 °C until to become the dried activated carbon. Finally, the black colour of activated carbon fiber was obtained. The experimental diagram of synthesis process of AC is presented in Figure 2.

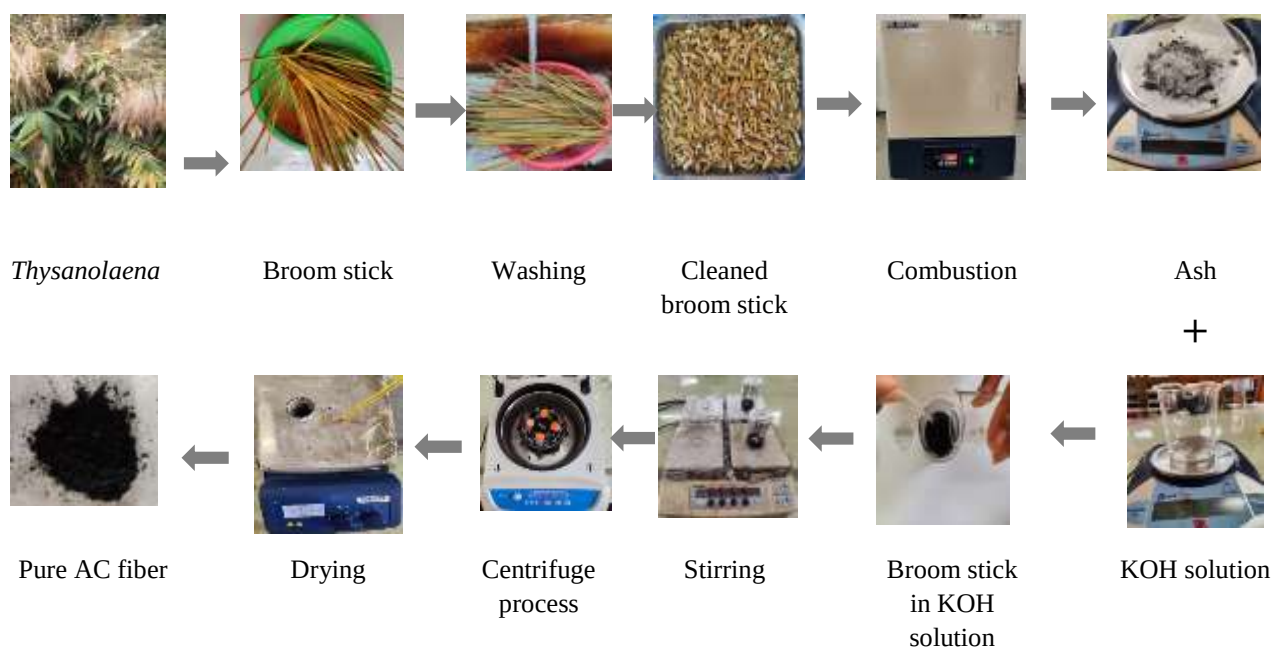


Figure 1 Synthesis process of activated carbon fibers by two-steps chemical activation process

Results and Discussion

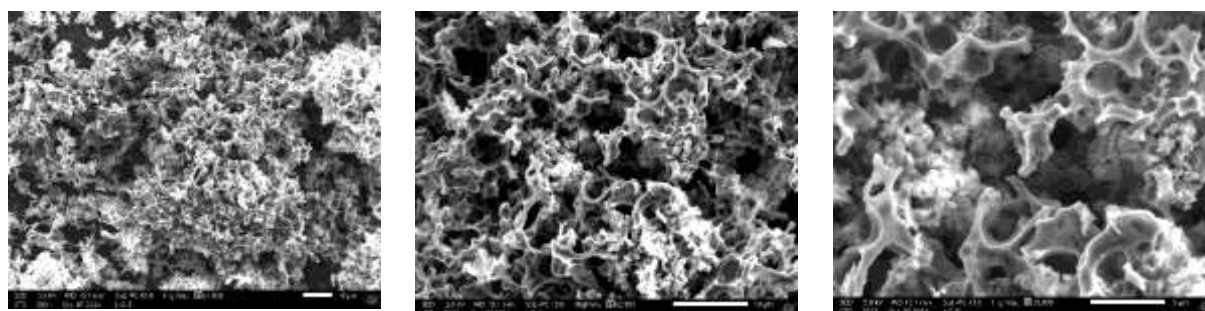


Figure 3 SEM image of broomstick synthesized by one-step chemical activation method at 600 °C at different magnifications

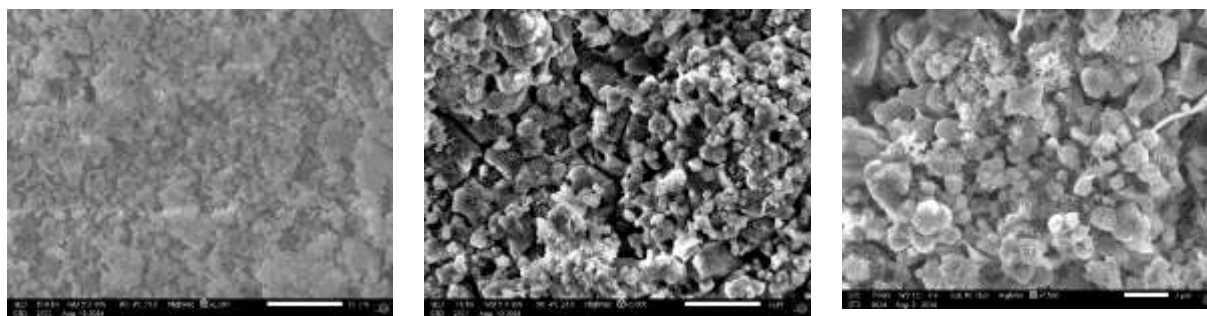


Figure 4 SEM image of broomstick synthesized by one-step chemical activation method at 700 °C at different magnifications

SEM analysis is carried out in order to observe the surface morphology of final products depending on two different activation processes. The colour of carbon-based materials is generally black. However, the colour of final product activated by one-step method is not black. This is due to the introduction of more functional groups which can change the light absorption and reflection properties of the biochar (Rajat Kumar Sharma et al., 2022). Figure 3 and 4 exhibits the SEM images of AC activated at 600 and 700 °C. The pore structure can be clearly seen in SEM image in broom stick activated at 600 °C. The surface morphology AC showed a rough texture with numerous cavities and channels. In contrast, there was no porous structure in SEM image of biomass activated at 700 °C. The surface of the AC was also more heterogeneous and less uniform.

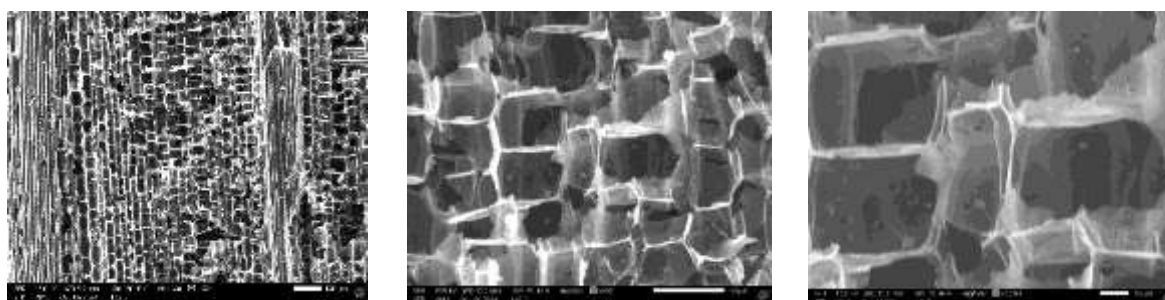


Figure 5 SEM image of broom stick synthesized by two-step chemical activation at 600 °C at different magnifications

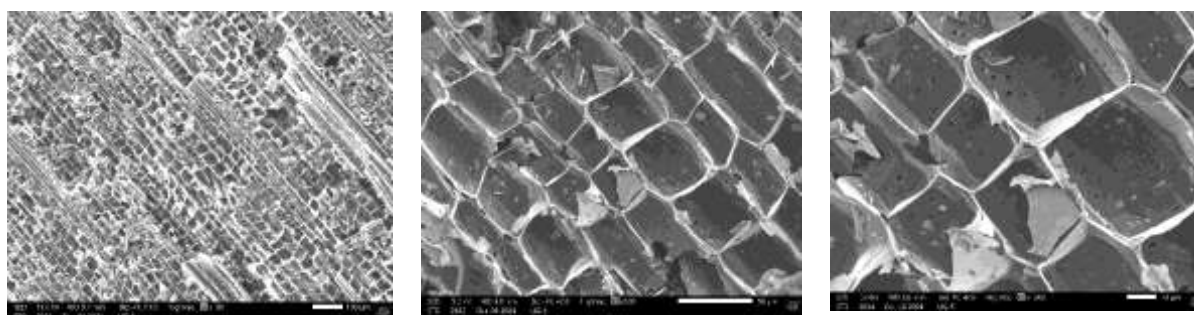


Figure 6 SEM image of broom stick synthesized by two-step chemical activation at 700 °C at different magnifications

The SEM image of AC prepared by two-step activation process is illustrated in Figure 6 and 7. The porous fiber structures of AC were clearly revealed in this image since KOH activation created a highly porous structure by etching the ash surface. Besides, the deep black color of AC was resulted due to the high-temperature in the combustion process of broom stick. In other words, the extensive carbonization and the removal of non-carbon elements during the combustion of biomass. Besides, the fibers were well-aligned and oriented in one direction. Thus, this structure has a possibility to support the better pathways for electron transport. Moreover, the well-connected pores ensure that ions can move freely throughout the electrode material which in turn improve the both of energy and power density. The average pore size is estimated by image J software. The average pore sizes were found out that 30.54 and 48.74 μm for 600 and 700 $^{\circ}\text{C}$ respectively. The pore size was become smaller when the ash temperature is increased from 600 to 700 $^{\circ}\text{C}$. As the pore of fiber is microporous type, ion transport of the anode in supercapacitor within the porous structure is likely to be enhanced (Zhao *et al.*, 2020). As a result, it is possibility to reduce the resistance and thus, it can enhance the charge/discharge rates as well as can contribute to higher power density in super capacitor (Mostafa S. Sayed *et al.*, 2024). Although, microporous fiber can improve the ion transport, overall capacitance will be contributed when they are compared to micropores and mesopores due to their lower surface areas in fiber.

Conclusion

The surface morphology and structure of broom stick-based activated carbon are investigated for the application of anode materials in supercapacitor. AC was prepared by chemical activation method. In one-step method at 600 $^{\circ}\text{C}$, the AC with porous structure was obtained. On the other hands, no porous structure was observed at activation temperature 700 $^{\circ}\text{C}$. In addition, the final product in one-step method could be formed only rough and inhomogeneous particles. In contrast, the AC with porous and aligned fiber structure were observed in two-step method at both 600 and 700 $^{\circ}\text{C}$. The pore size estimated by image J software was also regarded that in the microporous range. Thus, a larger pore area will allow for more active sites for charge storage, enhancing the capacitance. This fiber morphology may promote better wettability and contact with the electrolyte and can improve the overall electrochemical performance in supercapacitor. In conclusion, among the different surface morphology of AC depending on activation methods, AC with hierarchical pore structures has a possibility to improve both energy and power density in supercapacitor application.

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